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INVESTIGATION OF THE ULTRASONIC DYNAMIC
VISCOELASTIC PROPERTIES OF AQUEOUS
POLYETHYLENE OXIDE SOLUTIONS

ROBERT JAMES PRATHER, JR.


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INVESTIGATION OF THE ULTRASONIC
DYNAMIC VISCOELASTIC PROPERTIES
OF AQUEOUS POLYETHYLENE OXIDE
SOLUTIONS

by

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ABSTRACT

Some of the viscoelastic properties of aqueous solutions of polyethylene oxide were studied using the method of pulsed torsional wave propagation in the ultrasonic frequency range. Measurements of the changes in attenuation and phase of the wave train in a rod when the rod is immersed in the solutions permit calculation of the components of the complex shear modulus (or the complex viscosity). The results from this study are combined with those of others at different frequencies to obtain a measure of the variation of the viscoelastic properties over a wide frequency range. This can be interpreted qualitatively in terms of a distribution of relaxations at low frequencies corresponding to the configurational relaxation of the molecule as a whole and a relaxation frequency of approximately 40 kHz corresponding to that of the shortest chain segment. The real part of the complex viscosity approaches a value at high frequencies, dependent upon the concentration, which is greater than that of water thus showing the existence of a "local viscosity" which is dependent upon concentration.

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1. Introduction

Solutions of polyethylene oxide display both viscous and elastic behavior when subjected to a shearing stress thus they belong to the class of fluids called viscoelastic. The solutions also show a "non-Newtonian" viscous property called "pseudoplasticity", which is the designation given fluids that demonstrate an apparent decrease in viscosity with increasing rates of shear (1,15).

Polyethylene oxide is known to form very long linear chains in aqueous solutions and has the molecular structure $(-\text{CH}_2-\text{CH}_2-\text{O}-)_n$. The resin used to make the solutions was produced by the Union Carbide Company and has the trade name POLYOX-301. This polymer has an average molecular weight of about 4,000,000 (14). Henceforth in this paper the term polyox will be used vice polyethylene oxide when referring to this product.

Polyox, being a very long chain molecule, is flexible and can take up many different shapes. Since certain coiled shapes are more probably than others, the molecule will tend to return to those shapes if the stress is removed (7). This type of elasticity is called configurational elasticity, and the rate with which it regains the most probably shape after a deformation determines the relaxational time for such configurational elasticities.

Previous research at this institution (1,4) and at the University of Wisconsin (16) show that at frequencies in the mHz range the viscous effects of the polymer chains were relaxed while at frequencies in the range of 0.1-100 Hz the relaxation processes were present and must also be occurring at both higher and lower frequencies. Since this could not be explained in terms of the simple Maxwellian element

with a single relaxation frequency, this investigation was conducted at frequencies in the range of 20-70 kHz in order to obtain a measure of the distribution of relaxation frequencies.

2. Theory of Measurement

In order to investigate the viscoelastic properties of polyox solutions in the frequency range of 20-70 kilohertz, the method of pulsed torsional wave propagation was chosen (11). Piezoelectric torsional transducers cemented to a metal rod were used to send a train of torsional waves down the rod and to receive echos from the free end. The effects of the fluid in contact with the surface of the rod on the attenuation and phase of the wave were observed.

The solution of the Navier-Stokes equation for parallel oscillatory flow near a flat plate is given (13) as

$$U_y = U_0 \exp(i\omega t - \delta z)$$

where y is the direction of motion and z is the direction perpendicular to the plane, U_0 is the initial amplitude, ω is the frequency of oscillation and δ is a spatial decay constant given by

$$\delta = \sqrt{\frac{i\omega\rho}{\eta}}.$$

For the solutions of polyox being investigated the viscosity is always less than one poise, the frequency is greater than ten kHz and the density is approximately that of water. Thus a shear wave at a frequency of ten kHz is attenuated to a value of $1/e U_0$ in approximately 0.006 centimeters from the surface of the rod. Since the shear wave is attenuated in such a small distance compared to the radius of the rods used (0.635 centimeters), the assumption of plane waves is justified in formulating a working equation. However it also makes it

necessary to measure the viscoelastic properties by observing the loading effect of the fluid on the rod rather than by direct measurements. The viscous force the fluid exerts on the rod is given from the basic definition of viscosity as

$$F_y = S \eta \left(\frac{\partial U_y}{\partial z} \right)_{z=0} ,$$

where S is the surface area in contact with the fluid. The specific acoustic impedance Z for shear waves is defined by

$$Z = \frac{F/S}{U_y} ,$$

which, upon making the substitutions and dropping the time dependence becomes

$$Z = \sqrt{i \omega \rho \eta}$$

If R and X are the specific load resistance and reactance, Z can be written as

$$Z = R + i X$$

Gemant (3) proposed that η be written as

$$\eta^* = \eta_1 + i \eta_2$$

where η_1 is the normal flow viscosity coefficient and η_2 is an elastic viscosity term which vanishes for normal fluids. Substituting η^* for η gives

$$Z = R + i X = (\pi f \rho \eta^*)^{1/2} (1 + i) .$$

Squaring and separating the real and imaginary parts gives

$$\eta_1 = \frac{2RX}{\omega \rho} ,$$

$$\eta_2 = \frac{R^2 - X^2}{\omega \rho} .$$

Thus if R and X can be measured, η_1 and η_2 can be calculated.

For torsional waves propagating down radially symmetric rods the tangential particle displacement Θ is given by

$$\Theta = J_1(ka) \exp(i\omega t - \Gamma z)$$

where

$$k^2 = \frac{\rho\omega^2}{\mu} + \Gamma^2$$

and μ is the shear modulus of the rod and the wave is traveling in the +z direction with a propagation constant

$$\Gamma = A + iB$$

where A is the attenuation in nepers per centimeter and B is the phase shift in radians per centimeter (8). The specific acoustic impedance at a radius a is given by

$$Z = \frac{-T_{r\theta}}{\frac{\partial \Theta}{\partial t}} = \frac{\mu \left(\frac{\partial \Theta}{\partial r} - \frac{\Theta}{r} \right)}{\frac{\partial \Theta}{\partial t}}$$

which becomes for this case,

$$Z = \frac{i\mu k}{\omega} \left[\frac{J_0(ka)}{J_1(ka)} - \frac{2}{ka} \right].$$

Since by suitable choice of parameters the term ka can be kept small in the expansions of the Bessel functions the higher order terms may be neglected, giving

$$Z = \frac{i\mu a k^2}{4\omega}.$$

Assuming that the loading of the rod without the fluid is zero, then k must be zero and

$$\Gamma^2 = -\frac{\rho\omega^2}{\mu} = (A_0 + iB_0)^2$$

For metals A_0 is very much smaller than B_0 and the terms $2A_0B_0$ and A_0^2 are neglected. Thus

$$B_0 = \omega \sqrt{\frac{\rho}{\mu}} = \frac{\omega}{V_0},$$

where V_0 is the velocity of propagation of the wave in the rod.

Surrounding the rod with a fluid gives

$$k^2 = \frac{\rho \omega^2}{\mu} + \Gamma^2 = (A_0 + i B_0)^2 + (A + i B)^2.$$

Since $(B_0 + B) \gg (A_0 + A)$ in the usual case this becomes

$$k^2 = (B_0 + B)(-\Delta B + i \Delta A) \doteq 2 B_0 (-\Delta B + i \Delta A)$$

where ΔB and ΔA are $(B - B_0)$ and $(A - A_0)$.

Substituting, the impedance becomes

$$Z = \left(\frac{\rho V_0 a}{2} \right) (\Delta A + i \Delta B),$$

but since the observed attenuation and phase change are due to the total length of wave travel, the observed changes must be divided by the length $2nl_0$, where n is the number of the echo used and l_0 is the length of rod covered by the fluid. This gives

$$(1) \quad Z = \left(\frac{\rho V_0 a}{4n} \right) \left(\frac{a}{l_0} \right) (\Delta A' + i \Delta B')$$

where $\Delta A'$ and $\Delta B'$ are now the total change in the attenuation and the phase resulting from immersing the rod in the fluid. These are readily measurable quantities.

Instead of determining the complex viscosity, an alternate determination of the complex shear modulus G^* given by

$$G^* = G' + i G'' = \frac{\eta^*}{i\omega}$$

could be made.

3. Measurements

The velocity of propagation of the wave in the rod was determined by measuring the frequency change necessary to give a phase change of 2π radians since

$$V_0 = \frac{2Lf}{m + 9/4\pi}$$

where l is the length of the rod, m is the integer number of 2π radian shifts, and θ is the phase change upon reflection (10). The number of phase shifts m is given by

$$m = \frac{f}{\Delta f_1}$$

where Δf_1 is the frequency change required for a shift of 2π radians. The velocity of propagation can be determined from the slope of a plot of m as a function of frequency. The velocity V_0 was found to be slightly different for each rod since the rods were made from different stock. This variation was less than 0.8 percent. The observed velocity for each rod was used for calculations involving that rod.

All rods were checked using Newtonian fluids in the expected viscosity range of the polyox solutions to determine if the fluids had the expected loading effect on the rod as given by equation (1). It was found that the loading of the fluid on the rod was consistently within the experimental accuracy as long as the rods were highly polished. Since polyox solutions are basic, the use of aluminum rods prevented prolonged immersion in the polyox solutions. To avoid any increase in the loading due to the corrosion of the polished surface of the rods due to chemical reaction, the rods were repolished between successive runs and the time of submersion in the solutions was held to a minimum. The rods were also checked using standard fluids from time to time to insure that there was no degradation due to the repeated polishing.

It was not possible to measure with sufficient accuracy the loading effect of the fluids on the rods using the first returned echo. The echo train held the pulse shape until the fourth or greater echo so the third echo was used to give a greater attenuation accuracy.

The phase shift was measured by first calibrating the equipment in air to determine the frequency shift required to establish balance between successive 2π radian shifts of phase. Then surrounding the rod with fluid, the frequency shift required to re-establish balance was measured and converted into the phase shift in radians by

$$\Delta B' = \frac{2\pi \Delta f}{\Delta f_1}$$

Temperature control of the test fluid and the aluminum rod is needed because both the fluid properties and the velocity in the rod are temperature dependent. The entire system was enclosed in a water jacket with the temperature controlled to within $25.075 \pm .025$ °C.

The effect of this uncertainty on the velocity of the propagated wave pulse is 150 centimeters per second or 0.05 percent of the velocity. The effect on the phase of the wave pulse is 0.0056 radians or eight percent of the smallest observed phase shift.

The effect on the fluid properties is estimated to be less than 0.09 percent.

4. Equipment and Procedures

(a) Details of equipment

Following a method described by McSkimin (11), the equipment was constructed in the following manner.

The torsional transducers were made from one half inch barium titanate cylinders. Polarizing electrodes were placed as shown in Figure 2 and the crystals then heated in an oil bath to above the Curie temperature and 10,000 volts DC applied. After applying the voltage the crystal temperature was returned to room temperature with the voltage applied. Driving electrodes were then painted on the crystal using silver conducting paint as shown in Figure 2 (5). The

crystal frequency was then checked against computed values. This check also allowed a verification that only the torsional mode would be excited.

The rods were cut from standard one half inch aluminum bar stock and polished in a lathe using 600A sand paper followed by the use of automotive rubbing compound until a high polish was obtained. The crystals were then cemented to the rods using epoxy cement to give a hard connection.

For measurements the rods were supported inside the water jacket by needle pivot bearings at the top and bottom. The water jacket had an inside diameter of 3.3 centimeters. This distance is sufficient to prevent any wall effects. The water jacket was open at both ends to facilitate the insertion of the rod and the cleaning of the jacket between runs. The bottom was sealed with a plug containing the pivot bearing and a filling pipe. The top pivot bearing was held by an aluminum plate mounted by screws to nuts cemented to the jacket. A copper-constantan thermocouple, mounted in a thin wall copper tube, was inserted into the water jacket for measurement of the temperature inside the inner bore of the water jacket. The thermocouple was capable of measuring a temperature change of 0.025 °C and it was found that the temperature after equilibrium was established did not vary a measurable amount.

A block diagram of the equipment setup used for measurements is shown in Figure 1. The signal from the oscillator was split into two channels to provide a driving signal to the crystal and a direct reference signal for phase and echo amplitude measurements. The driving signal was pulsed by the toneburst generator giving a pulse of eight

or 12 cycles depending upon the frequency used. A second gate circuit triggered by the toneburst generator, was added to give additional isolation. The toneburst generator and gate combination gave -76 db isolation for the ratio of the cut off signal to the continuous signal. The pulse repetition rate of the toneburst generator was set at about two pulses per second, which allowed sufficient time for each echo train to be absorbed to below the noise level. The return echos were then amplified and mixed with the direct, attenuated signal from the oscillator. This signal was then observed on an oscilloscope triggered by the toneburst generator.

The direct reference signal was attenuated by a 600 ohm noninductive attenuator capable of measuring 0.1 db steps. Frequency was measured using a counter with a ten second count period.

All electronic equipment was operated using constant voltage generators. This was found necessary to give stable operation of the oscillator and to reduce the noise in the amplifier and toneburst generator. The oscillator was left in continuous operation with no load between measurements to insure minimum frequency shift due to thermal instability in the oscillator. The other equipment was turned on at least one hour prior to making measurements.

(b) Procedures

For changes in attenuation and phase, the mixed signal was displayed on the oscilloscope set on time and magnitude scales to give as large a display of the third echo as possible. With only air surrounding the rod, eight measurements of frequency and attenuation necessary to cancel the echo were made, from which an average value was obtained. The frequency shift required to give 2π radian phase

shift from each frequency reading was recorded. The temperature of the rod was measured and recorded. The preheated fluid was then forced into the inner bore of the water jacket using compressed air. This was found to be necessary as filling from the top created air bubbles which required a considerable time period to settle out. To avoid the possibility of the temperature not being constant across the tube, the thermocouple was arranged so that it could be placed next to the rod prior to a run and then shifted away from the rod during the acoustic measurements. When thermal equilibrium was reached, the measurements of frequency and attenuation to give echo cancellation were repeated. The change in frequency and attenuation were converted into changes of phase in radians and attenuation in nepers. Using these values of changes, the resistive and reactive loading could then be computed. At least two separate measurements using different fluids of the same concentration were made and these measurements averaged to give the final value of acoustic loading due to the fluid.

The smooth curves of the acoustic loading were then used to obtain values of resistance and reactive loading to be used in computing the terms of the complex viscosity and shear modulus.

(c) Solution mixing procedure

The solutions were mixed using a low speed variable speed mixer with a maximum speed of 200 rpm. The speed of the mixer and the depth of the blade of the mixer were adjusted to give the water a vortex not quite touching the blade of the mixer. The polyox powder was then added with a shaker until the surface was saturated with powder. This was repeated as the polyox was drawn under the surface

by the vortex. By using this technique, large lumps of undissolved polyox could be avoided. After all the polyox was added to the water, the solution was allowed to mix until there were no particles of undissolved polyox suspended in the solution. The adding of the polyox and dissolving after all had been added took from several hours to two days, depending upon the concentration.

5. Results

The frequency range covered in the present study is too narrow to describe adequately the viscoelastic behavior of the polyox solutions as a function of frequency. However, when the present results are combined with the results obtained in a previous study by Kinnier (4), it is possible to get some measure of the frequency response. Figures 7 and 8 show the frequency response of the real parts of the complex viscosity and shear modulus.

The behavior of the real part of the complex shear modulus is very similar to that found by Mason (8) for solutions of polyisobutylene at higher frequencies. Since polyox is a longer molecule, the lower frequency was expected. Mason gave the interpretation of the steep rise in G' as the relaxation of the smallest chain segment. This interpretation seems to apply for polyox as it is the first relaxation that would involve all molecules since it does not depend upon the concentration or the length of the molecule. Below the frequency of this relaxation, G' decreases slowly as frequency decreases. Philippoff (6) describes this as the behavior of a fluid with a distribution of relaxation times. Since polyox molecules have a distribution of molecule lengths, it was expected that the relaxations dependent upon the length of the polyox molecule would also have a

distribution of frequencies.

The real part of the complex viscosity was found in this study to approach a steady value at the higher frequencies. Since the 0.25 and 0.50 percent solutions approached the same limiting value of 0.015 poises and the one percent solution approaches a higher value of 0.023 poises, the idea introduced by Peterlin and Signer (6) of a "local viscosity" which depends upon the interaction of the molecules with each other appears to hold for the polyox solutions.

6. Discussion of Error

Statistical uncertainty in the observed quantities ranged from a high of 17 percent to five percent. The higher values were for the measurements in the lower frequencies and lower concentrations. Smoothing of the observed data was accomplished by plotting the resistive and reactive loading versus frequency and taking the best fit of these points to compute the components of the complex viscosity and complex shear modulus. Thus the probably error is less than the statistical error and this can be observed in the smoothness of the transition from values observed in this study to the values found by Kinnier and Reister.

7. Acknowledgements

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Frequency (cps)	20144		30365		40268		50509		60655		70151	
	R	X	R	X	R	X	R	X	R	X	R	X
.25	33.5	31.9	40.9	39.1	46.6	44.1	51.1	47.5	55.2	50.3	58.9	52.5
.5	35.2	32.3	41.6	39.0	47.5	40.0	54.5	40.5	61.5	43.0	67.3	45.2
1.0	49.7	34.8	57.1	42.9	62.9	49.3	68.3	54.5	74.0	59.0	81.0	63.1

Table 1. Observed Values of the Resistive and Reactive Loading. The Resistive and Reactive Loading are in Dyno-sec/cm² and the Concentration in Weight Percent.

Frequency (cps)	20144		30365		40268		50509		60655		70151	
	G'	G''	G'	G''	G'	G''	G'	G''	G'	G''	G'	G''
.25	110	2130	260	3110	190	3950	250	4730	610	5480	720	6200
.5	196	2280	210	3251	657	3807	1333	4423	1937	5290	2491	6096
1.0	1272	3466	1443	4918	1539	6892	1695	7446	2014	8761	2601	10,255

Table 2. Real and Imaginary Components of the Complex Shear Modulus. G' and G'' are in Dynes/cm² and Concentration in Weight Percent.

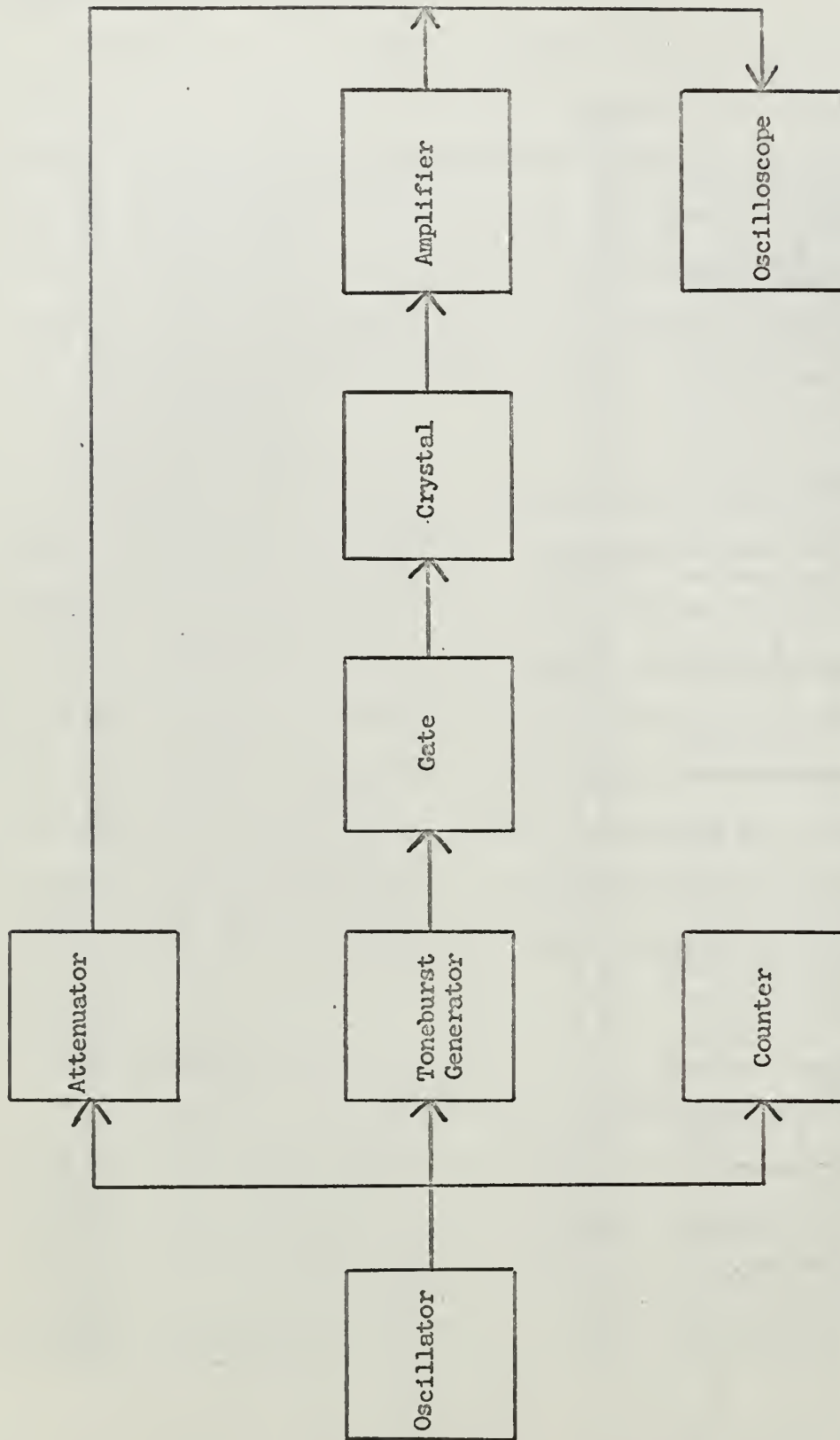
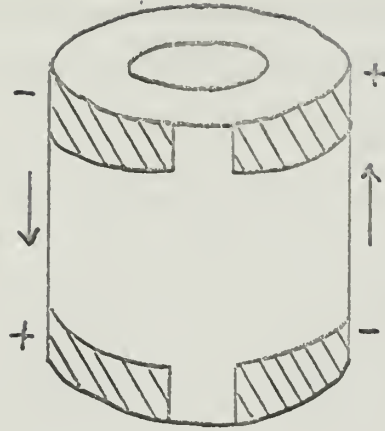
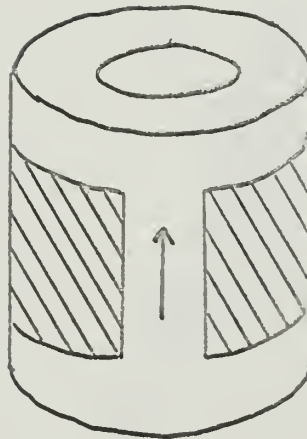


Figure 1. Block Diagram Showing Experimental Setup for Measurements Using Traveling Torsional Waves.



(a)



(b)

Figure 2. Barium Titanate Crystal Electrode Location for (a) Polarization and (b) Driving.

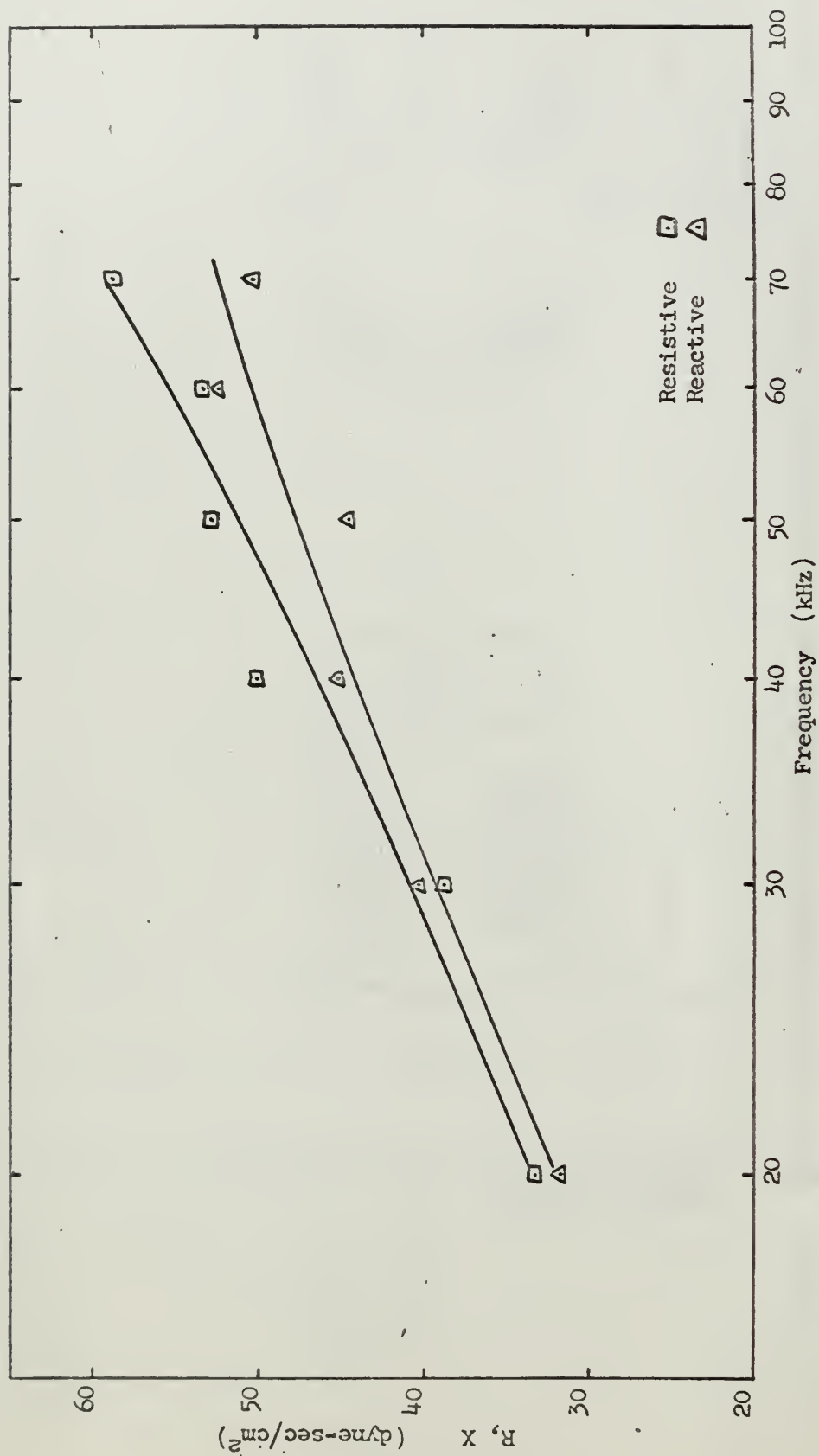


Figure 3. The Resistive and Reactive Loading for 0.25 Percent Polyethylene Oxide as a Function of Frequency.

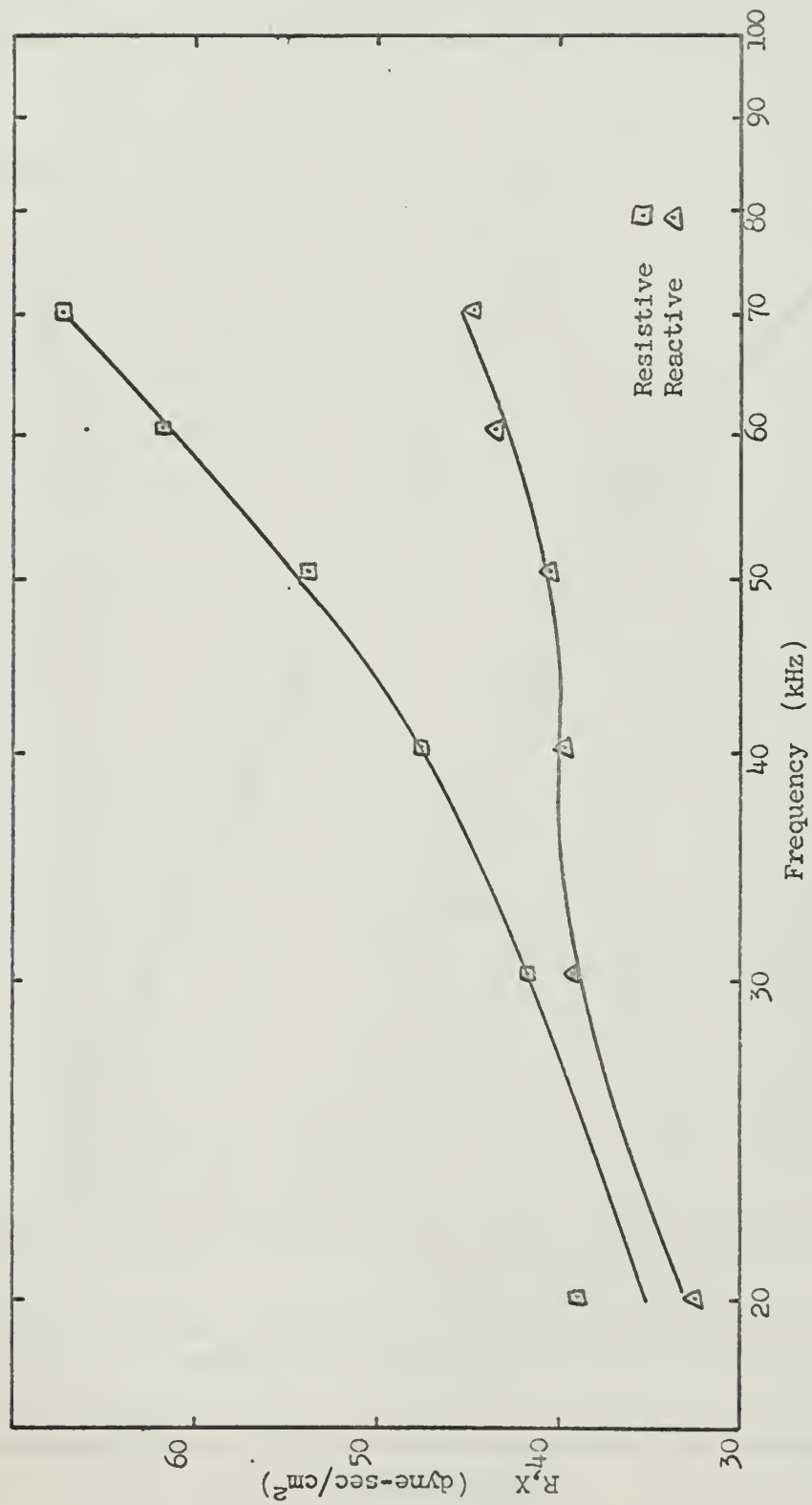


Figure 4. The Resistive and Reactive Loading for 0.5 Percent Polyethylene Oxide as a Function of Frequency

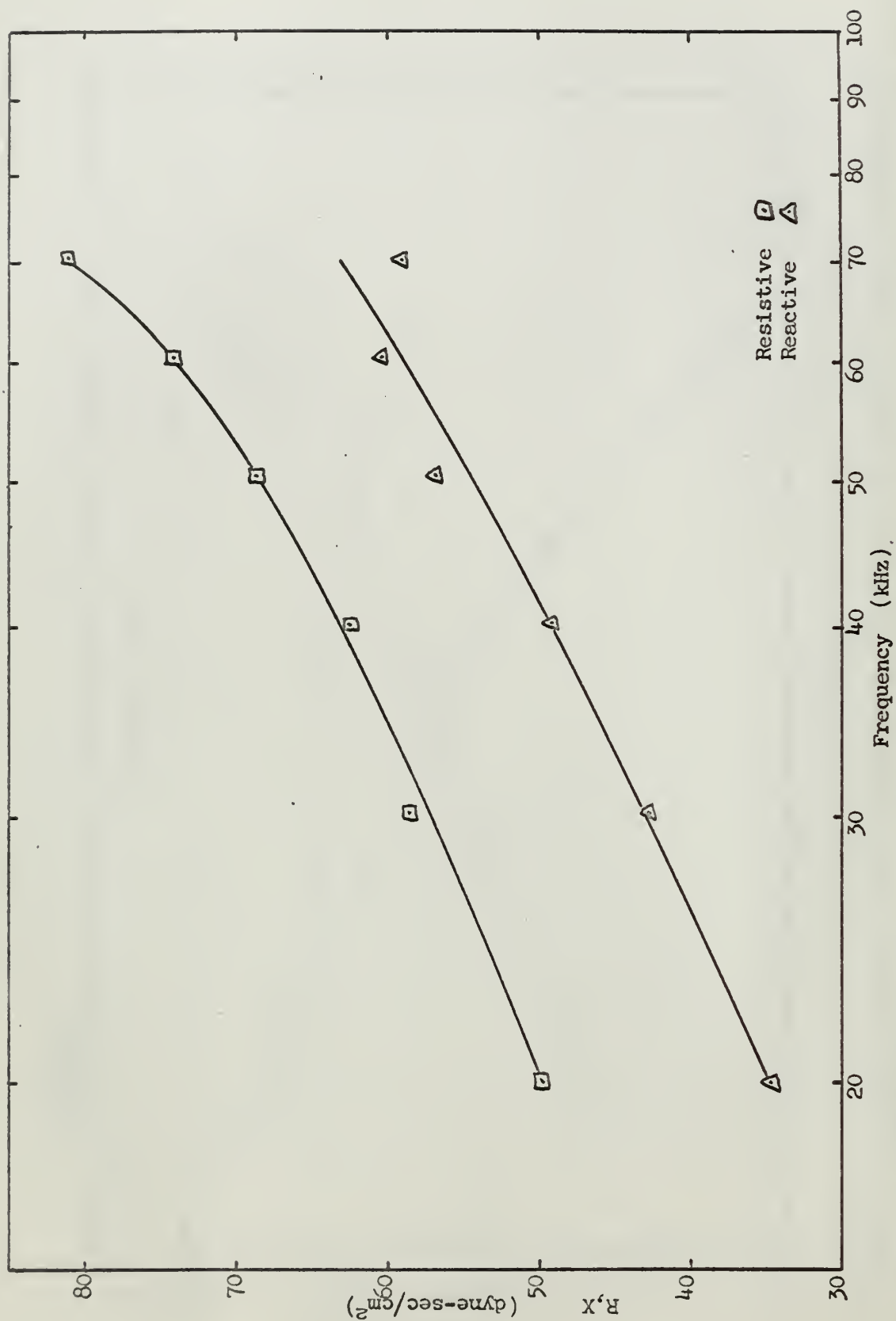


Figure 5. The Resistive and Reactive Loading for One Percent Polyethylene Oxide as a Function of Frequency.

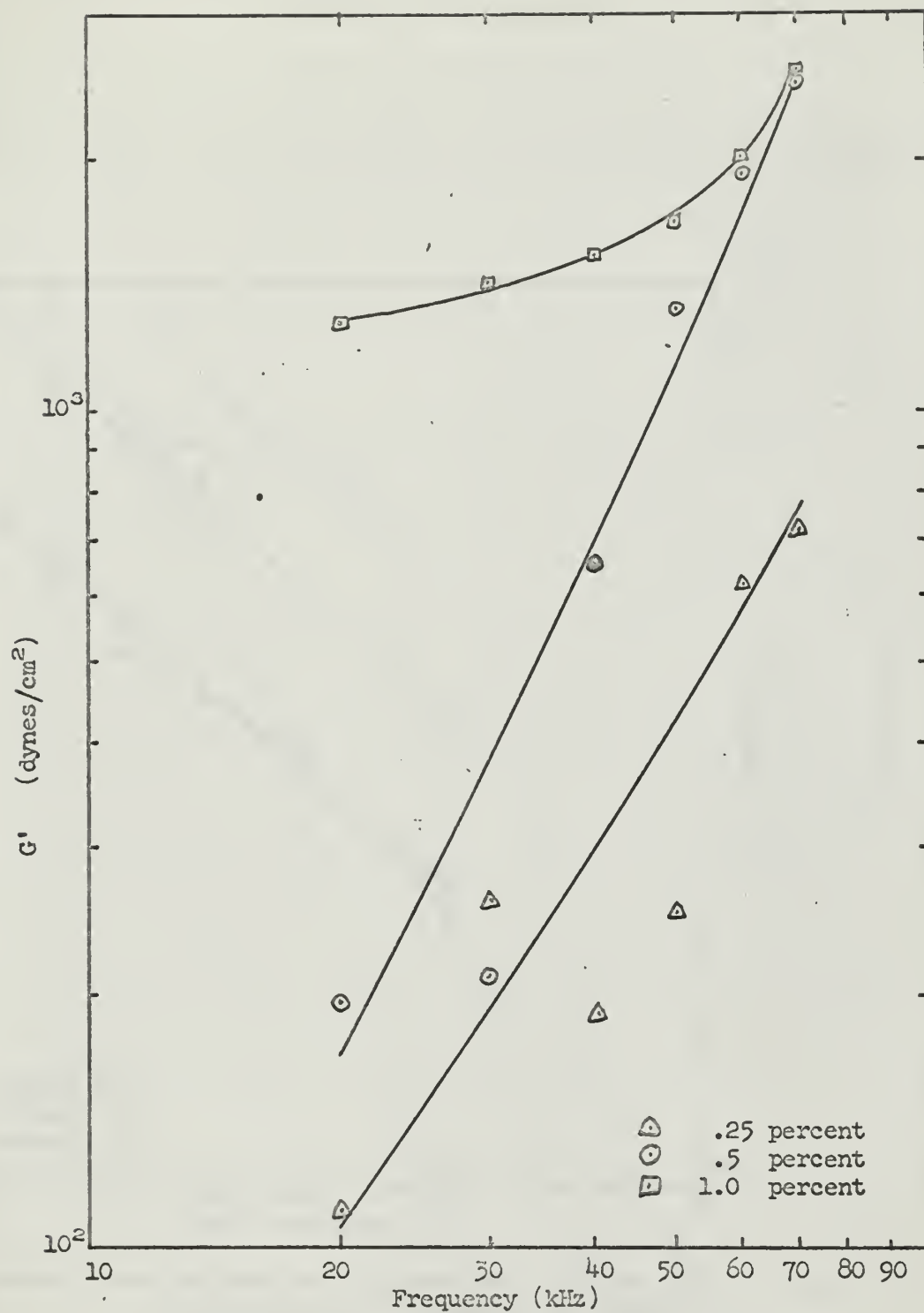


Figure 6. The Real Component of the Complex Shear Modulus as a Function of Frequency and Concentration.

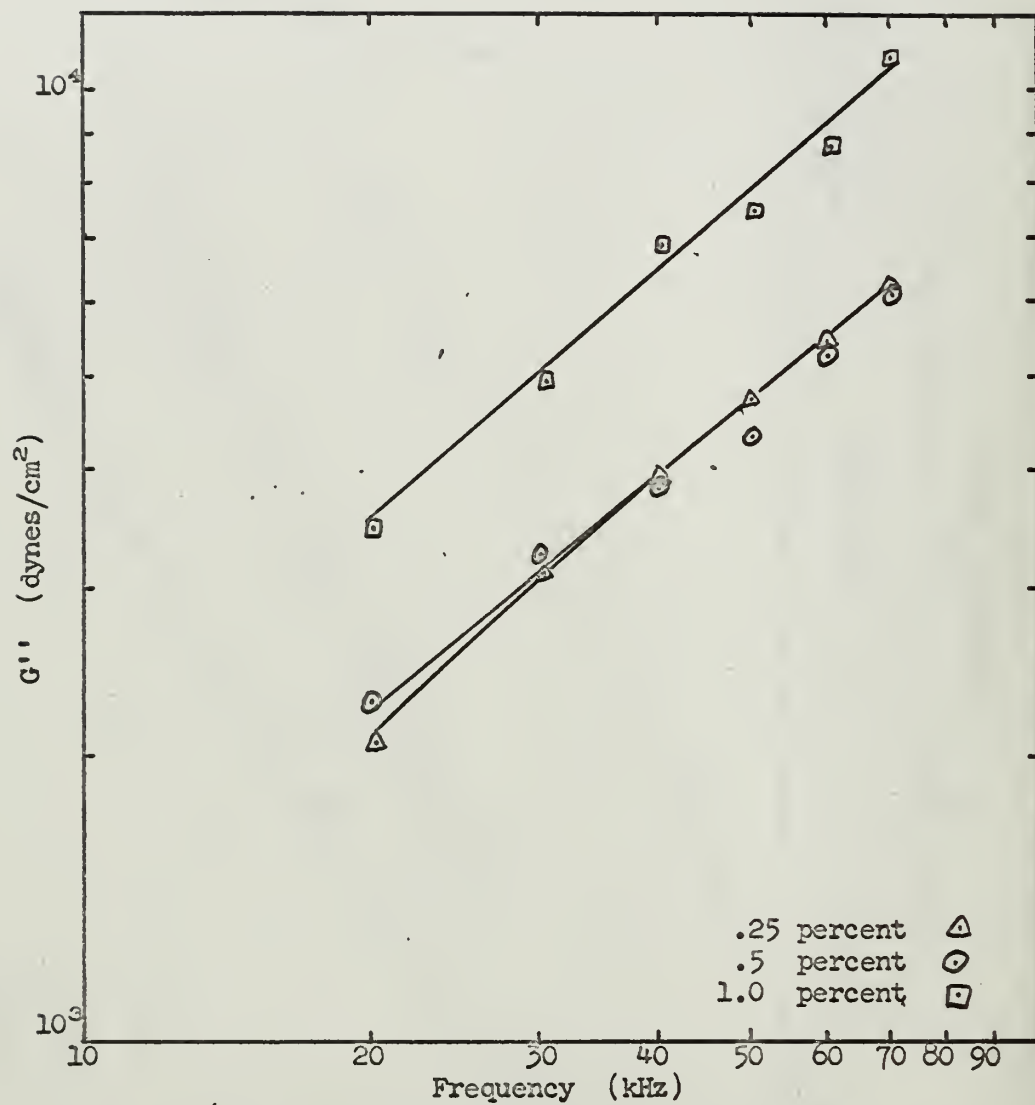


Figure 7. The Imaginary Component of the Complex Shear Modulus as a Function of Frequency and Concentration.

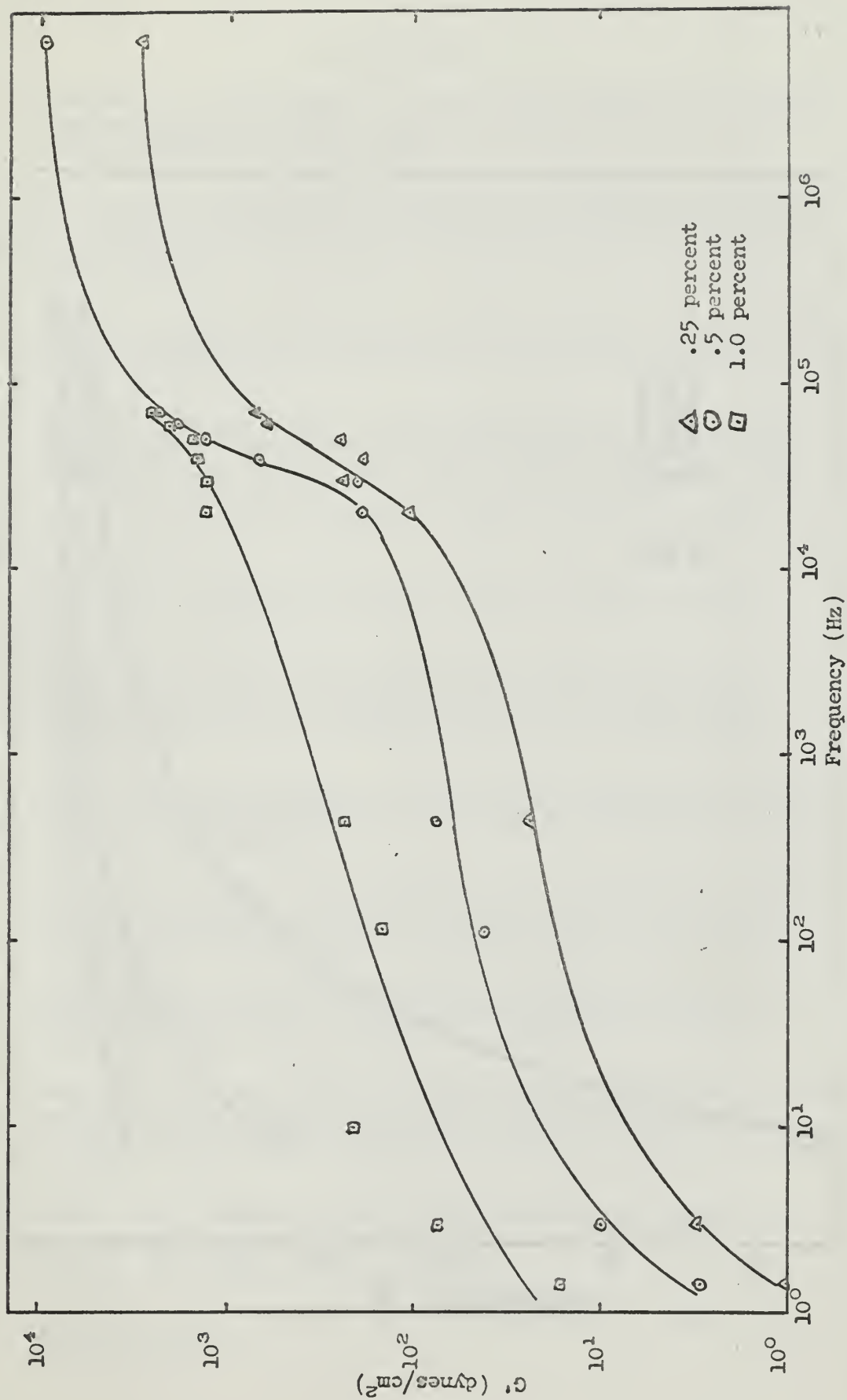


Figure 8. The Real Component of the Complex Shear Modulus as a Function of Frequency and Concentration.

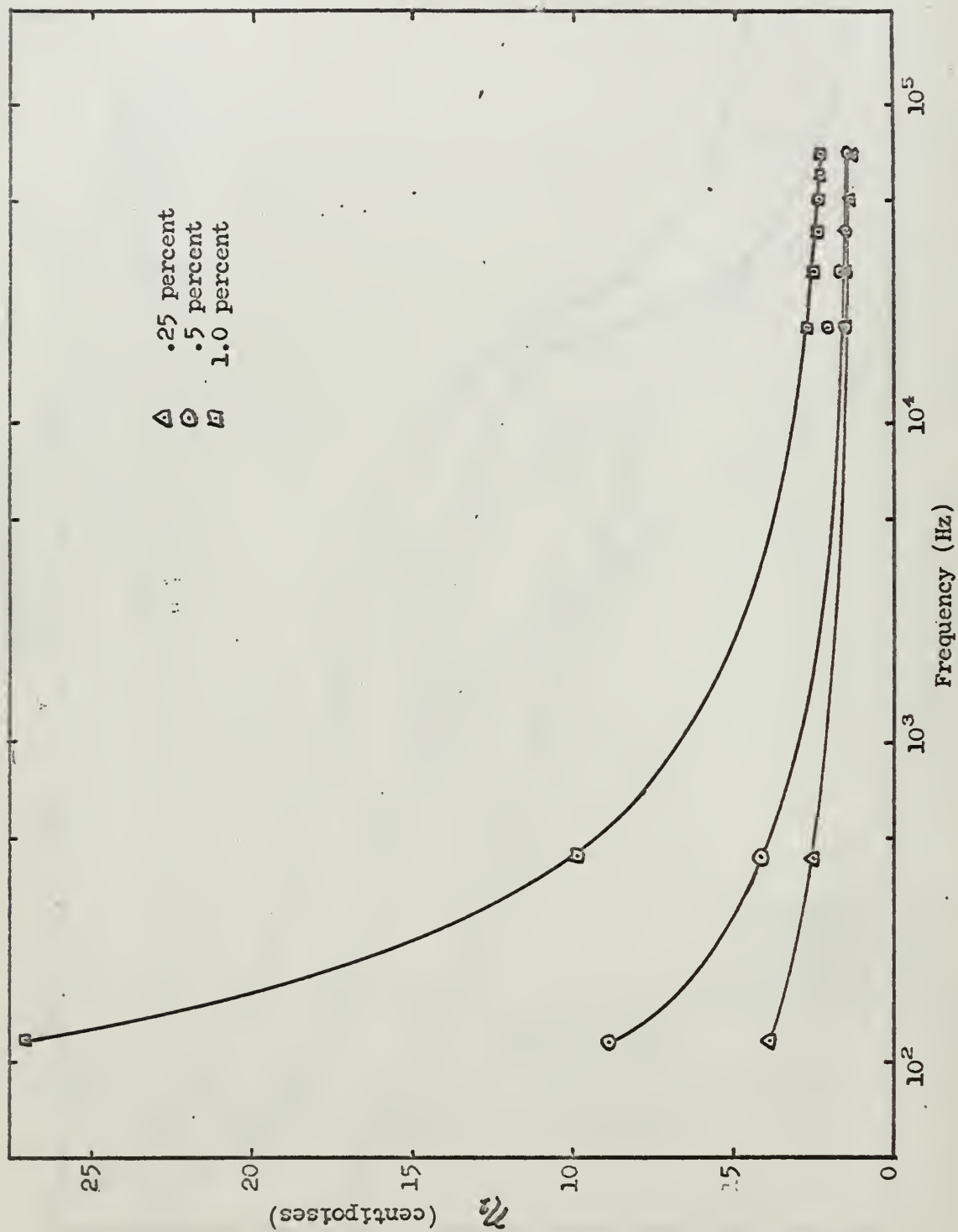


Figure 9. The Real Component of the Complex Viscosity as a Function of Frequency and Concentration.

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13. ABSTRACT Some of the viscoelastic properties of aqueous solutions of polyethylene oxide were studied using the method of pulsed torsional wave propagation in the ultrasonic frequency range. Measurements of the changes in attenuation and phase of the wave train in a rod when the rod is immersed in the solutions permit calculation of the components of the complex shear modulus (or the complex viscosity). The results from this study are combined with those of others at different frequencies to obtain a measure of the variation of the viscoelastic properties over a wide frequency range. This can be interpreted qualitatively in terms of a distribution of relaxations at low frequencies corresponding to the configurational relaxation of the molecule as a whole and a relaxation frequency of approximately 40 kHz corresponding to that of the shortest chain segment. The real part of the complex viscosity approaches a value at high frequencies, dependent upon the concentration, which is greater than that of water thus showing the existence of a "local viscosity" which is dependent upon concentration. (U)			

14.

KEY WORDS

Polyethylene Oxide
Non-Newtonian Fluid
Viscoelastic Fluid

LINK A

LINK B

LINK C

ROLE

WT

ROLE

WT

ROLE

WT

INSTRUCTIONS

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